Integrating Hazard Identification and Risk Assessment into Course-based Undergraduate Research

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Integrating Hazard Identification & Risk Assessment into the Curriculum

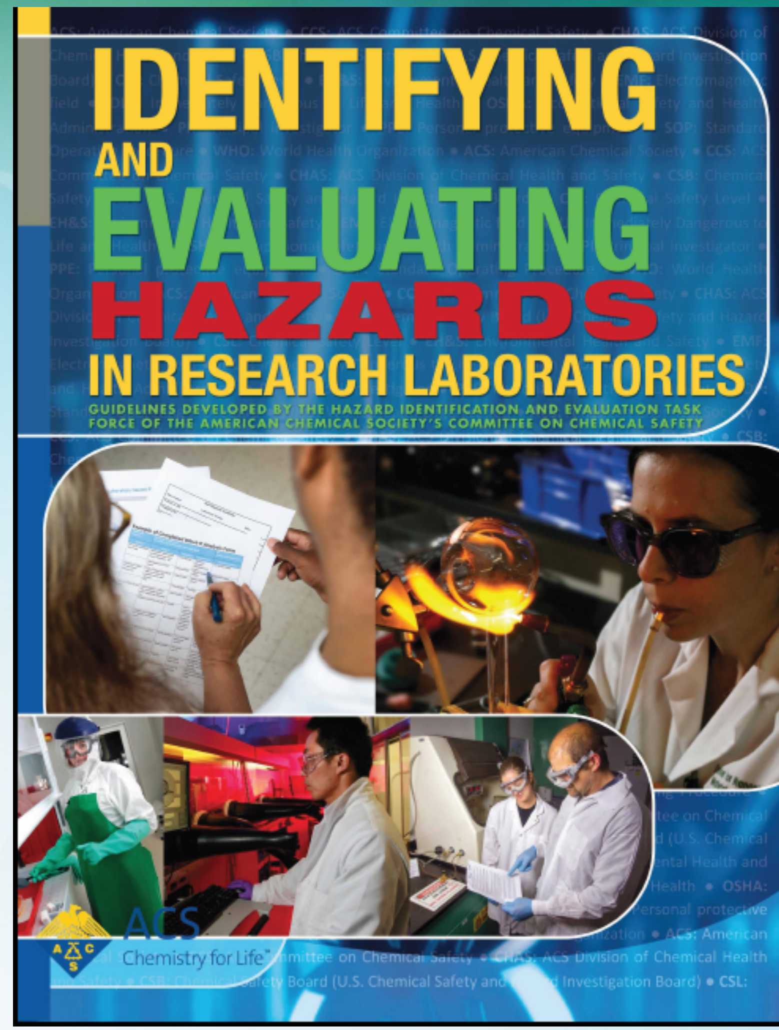
- Background – Teaching safety beyond rules
- Course structure – Inorganic Lab (CHE 3405)
- The independent research project – Synthesize an acetylacetonate (2,4-pentanedione) / transition metal coordination compound
- Recognized opportunity – Student engagement in safety
- Improvement – Future directions
- Recognized opportunity – Improving a CURE

Teaching Hazard Identification & Risk Assessment

Background

The Chronology of This Journey

- **<u>2012</u>** – Work began on the ACS Publication *Identifying and Evaluating Hazards in Research Laboratories*[1]



[1] Hazard Assessment in Research Laboratories

- **<u>2014</u>** – Our department added a graded hazard identification and risk assessment assignment to our Capstone course – we used the "Job Hazard Analysis" (JHA)

  - A JHA, requires that students break their process into steps, identify hazards, estimate risk, implement controls to minimize risk, and indicate how to prepare for emergencies (RAMP)[4]

[4] Hill, R. H.; Finster, D. C. Laboratory Safety for Chemistry Students; John Wiley & Sons, Inc.: Hoboken, NJ, 2010; p 1-7.

- **2016** – We moved the assignment to our junior level “Introduction to Research” course (CHE 3000)
- **2016** The pedagogy used with the assessment results were published in the ACS Symposium Book Series – *Integrating Library and Information Literacy into Chemistry Curricula*[2]

[2]Sigmann, S.B.; McEwen, L.R. Teaching Chemical Hygiene and Information Skills Using Risk Assessment. In *Integrating Library and Information Literacy into Chemistry Curricula*; Lovitt, C.F.; Shuyler, K.; Li, Y, Eds; ACS Symposium Series; American Chemical Society: Washington DC, 2016; 1232, pp 57–92.

Inorganic Laboratory (CHE 3405)

Course structure

Inorganic Lab CHE 3405 Course Structure

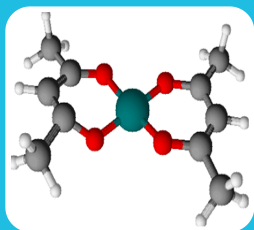
- Fifteen week semester w/ exam week
- There were two sections of 8 or 9 students in F16
- Typical inorganic synthesis methods taught based on ACS guidelines[3] (synthesis, purification, characterization)
- Additionally – There is an embedded, student driven, three week independent synthesis project that is ~40% of the grade

[3]ACS CPT Inorganic Chemistry Supplement (2015)

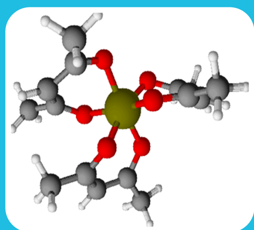
Synthesis of a M(acac)$_x$ Complex

The Independent Research Project

General Synthesis Reaction

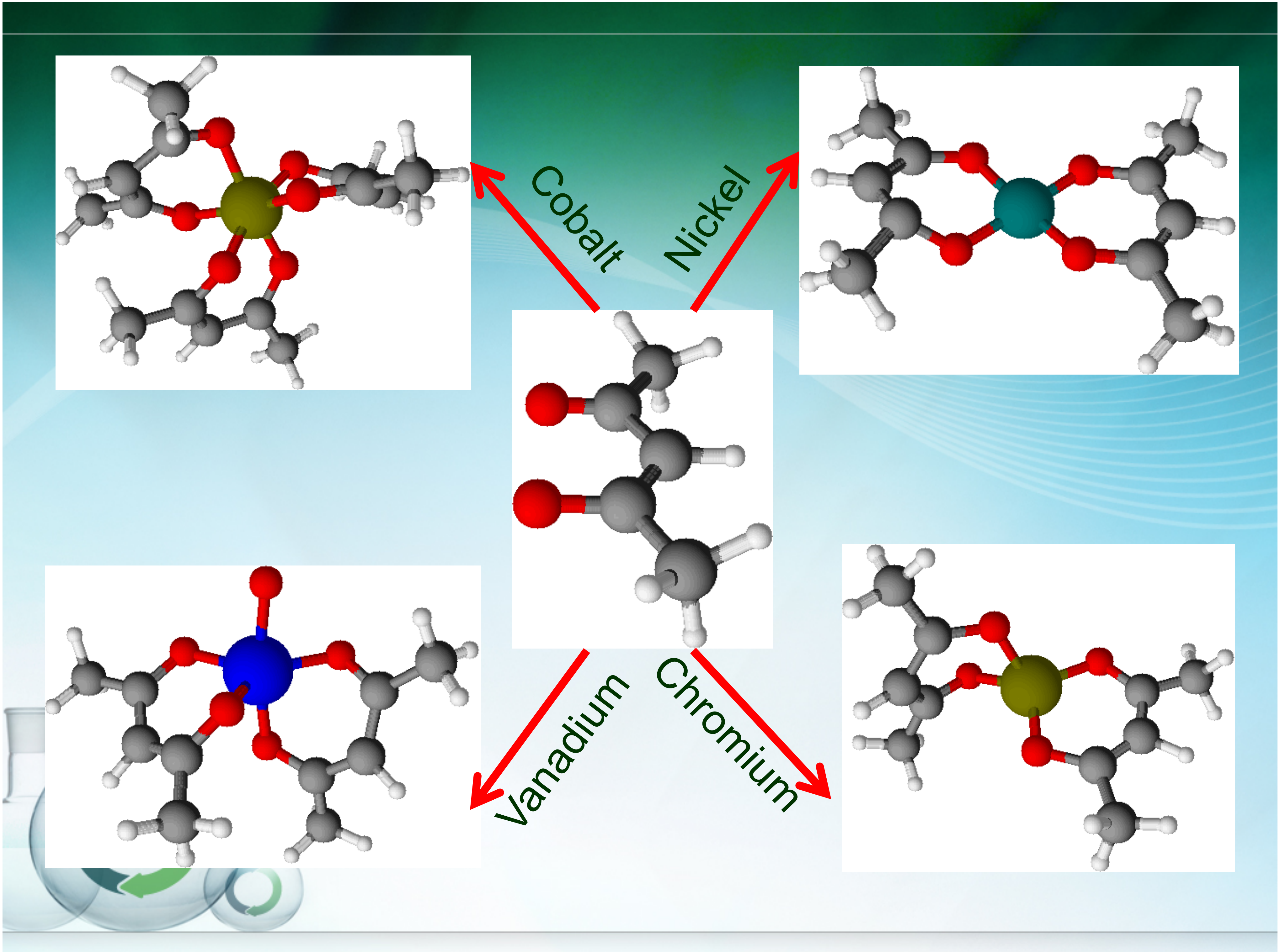


$$MX_2 + 2\ \text{acacH} \rightarrow M(\text{acac})_2 + 2HX$$



$$MX_3 + 3\ \text{acacH} \rightarrow M(\text{acac})_3 + 3HX$$

No two students in the same section can synthesize the same compound


Cobalt
Nickel
Vanadium
Chromium

The Project Outline – Library Assignment

- **<u>Literature Search</u>** – Use SciFinder, students must research aceylacetonate (structure, IUPAC name, CAS#, homoleptic definition, sources which describe synthesis of any metal compound with this ligand)
- Students must reference ***<u>primary</u>*** literature (no lab manuals, etc.)

The Project Outline – Reagent Form

- **Approved Reagent Form** – this ensures that the synthesis produces enough compound to characterize, but not a huge excess of waste
- Additionally, this assignment ensures that reagents are available in the stockroom
- An added benefit is that students learn to navigate the university inventory system when determining availability of chemicals
- Information required: compound to synthesize, theoretical yield (1-5 g), all reagents needed with amounts (from literature)

The Project Outline – Reagent Form

Name_____________________________ Compound to Synthesize___________________________

Theoretical Yield in Grams (must be between 1-5 grams unless previously approved by instructor – note that you may have to adjust amounts for this form)

____

All reagents needed except water must be listed on this form (remember that solvents are reagents). You must list the amount you need for your calculated theoretical yield. Do not, for example, list “acetone – enough to dissolve”.

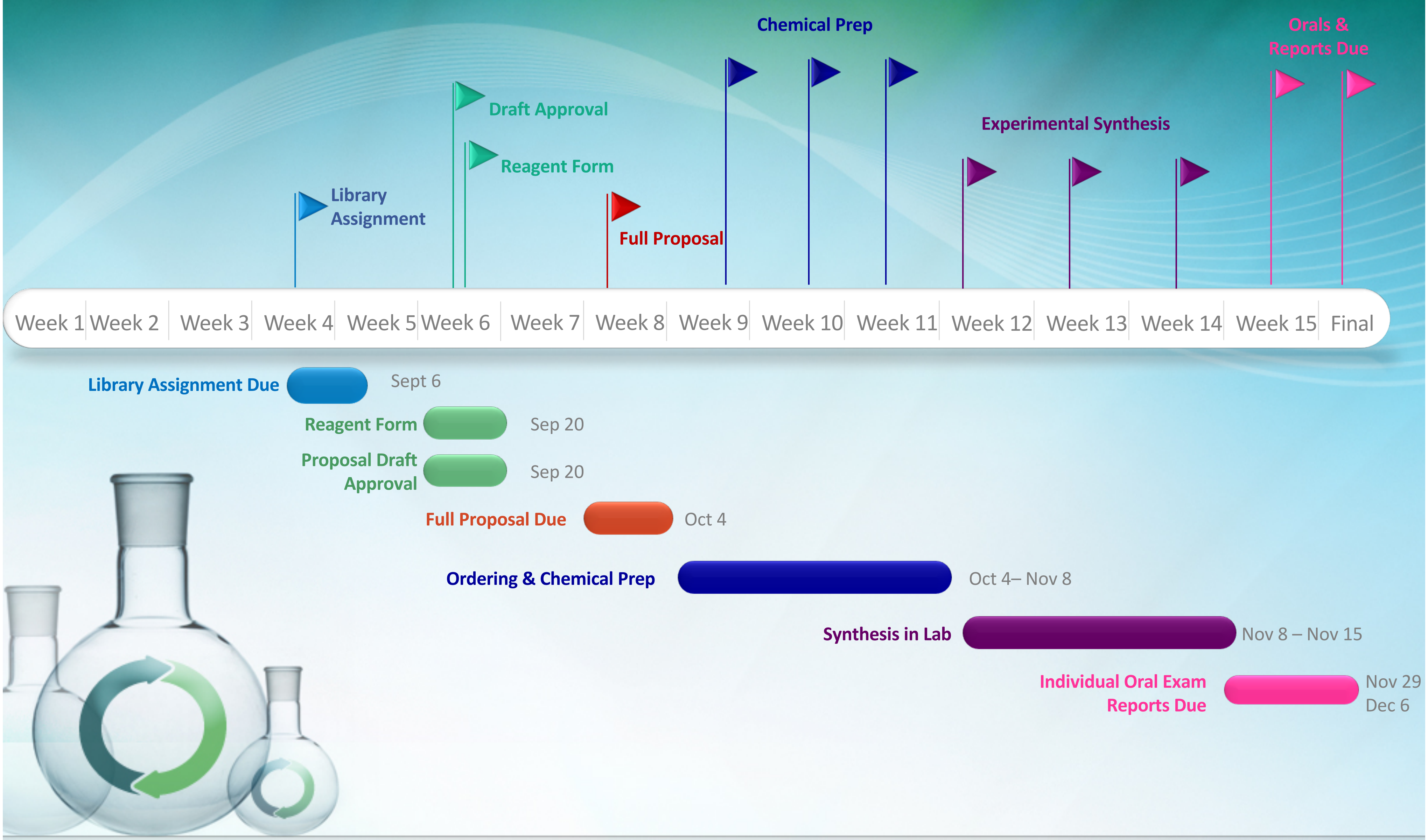
This form must be completed and signed by the CHE 3405 Instructor and the Stockroom Manager by the deadline listed in your syllabus. The CHE 3405 Instructor should sign the form first.

Reagent (grade/purity)	CAS Number	Amount

The Project Outline – Additional Components

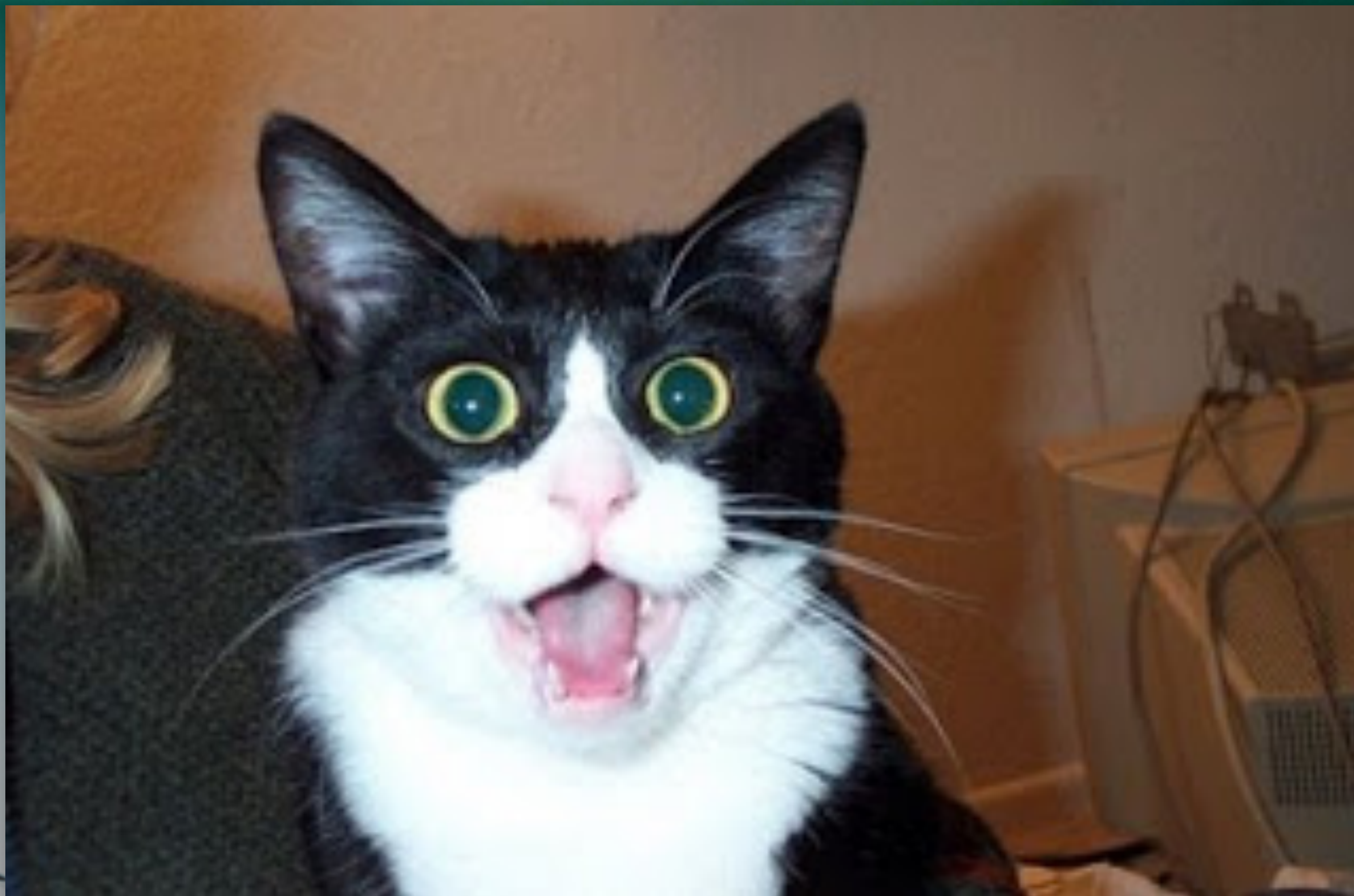
- **Research Proposal Outline** – This document includes the title page, abstract, introduction, experimental section, expected results, and primary references
- **Formal Research Proposal** - Based on approved outline
- **Research Paper** – Submitted in ACS research paper format
- **An Oral Presentation** (course exam) – One on one with the instructor

Inorganic Synthesis Project Timeline



Student Engagement in Safety

Recognized Opportunity



Chemical Hazards – Metal Compounds

Complex	CAS	Formula	8 hour TWA or Ceiling
cobalt(II) carbonate	57454-67-8	$CoCO_3 \cdot xH_2O$	0.02 mg/m^3
palladium(II) chloride	7647-10-1	$PdCl_2$	No Data
aluminum sulfate octadecahydrate	7784-31-8	$Al_2(SO_4)_3 \cdot 18H_2O$	2 mg/m^3
chromium(III) chloride	10025-73-7	$CrCl_3$	0.5 mg/m^3
copper(II) sulfate pentahydrate	7758-99-8	$CuSO_4 \cdot 5H_2O$	1 mg/m^3
nickel(II) nitrate hexahydrate	13478-00-7	$Ni(NO_3)_3 \cdot 6H_2O$	0.015 mg/m^3 0.10 mg/m^3
iron(III) chloride hexahydrate	10025-77-1	$FeCl_3 \cdot 6H_2O$	1.0 mg/m^3
vanadium(IV) oxide sulfate hydrate	123334-20-3	$VOSO_4 \cdot xH_2O$	0.050 mg/m^3 *
mercury(II) oxide	21908-53-2	HgO	0.025 mg/m^3
yttrium(III) nitrate hexahydrate	13494-98-9	$Y(NO_3)_3 \cdot 6H_2O$	1.0 mg/m^3
potassium permanganate	7722-64-7	$KMnO_4$	0.2 mg/m^3

NIOSH
NIOSH*Ceiling
ACGIH TLV

Chemical Hazards – Acetyl Acetone

BP 138 deg C
FP 34 deg C (Closed cup)

Warning

Incompatible with oxidizing materials
Flashback along vapor trail may occur
Above 34 deg C explosive vapor/air mixtures may be formed.

LD50 rabbit values (1370 (male), 790 (female), and 4870mg/kg bw),

LC50 (rat) = 1224ppm/4h

Odor Threshold 0.010 ppm

Danger

Vapor irritating to eyes. Liquid irritating to skin and eyes.

May cause damage to organs through prolonged or repeated exposure (central nervous system, thymus)

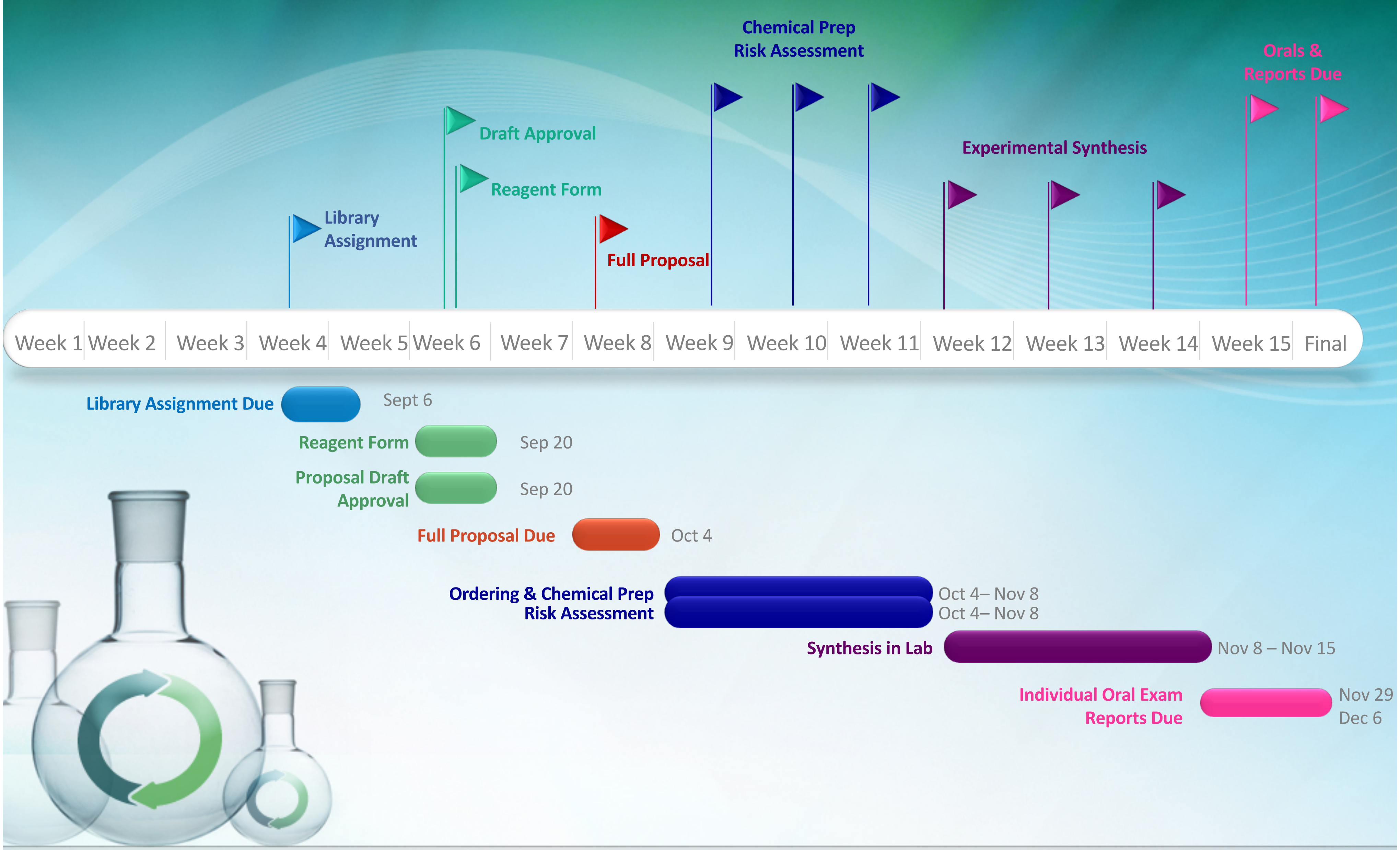
Warning

The Project Outline – Risk Assessment

"Hey, these guys learned how to do a risk assessment in CHE 3000.

Let's have them prepare a JHA for their independent project!"

Inorganic Synthesis Project New Timeline



Risk Assessment Process Used F16

- JHA draft submitted
- Draft Evaluated
- Scheduled meetings with each student for discussion
- Discussion comments used to improve tool and improved JHA resubmitted
- Tool approval ***required prior to the start of the independent project***

Excerpts – Student JHAs

Work Steps and Tasks Describe the tasks / steps involved in the work – in order	Hazards Identified for each Task / Step	Risk Level Risk Nomogram can be used	Control / Safe Work Procedures for each Task / Step Controls to be implemented
Step 1: Weight 1.002 g of $CrCl_36H_2O$ in 50- mL beaker	Inhalation, spill, dermal contact	Moderate risk	• Weigh in dust hood • Wear chemical splash goggles and nitrile gloves • Have a spill kit on location review use

13. Dissolve iron(II) chloride in water (100 mLbeaker)	Spillage Skin contact	Moderate	Work in spill tray to minimize potential of the spill Careful movement
14. Dissolve acetylacetone in methanol	Spillage Skin contact flammable	Moderate	Same as above, no open flames remove excess solvents from hood when complete with step

Step 17: Pour waste into Organic Waste container	Inhalation, dermal contact, spilling, improper mixing with nitric acid	Moderate risk	• The label of the container will be checked thoroughly before pouring in waste • The instructor will be asked to insure that the appropriate waste container is used • Both hands will be used during transfer • Chemical splash goggles as well as nitrile gloves will be worn

Hazards Recognized/Risk Reduced

By working with the students we were able to very efficiently minimize hazards

- All work was done on spill trays
- Physical hazards from chemicals and equipment identified
- All of the starting materials were manipulated in either a dust hood or fume hood
- Nitric acid/organic waste hazard was eliminated
- Correct glove material was identified
- Waste residues from the hazardous materials were minimized by eliminating transfers

Future directions – Iterate!

Improvement

Future Improvements – Reagent Form

Reagent (grade/purity)	CAS Number	Amount
vanadium(IV) oxide sulfate hydrate (ACS or reagent)	123334-20-3	5 g
2,4-pentanedione (distilled)	123-54-6	10 mL
Acetone (lab grade, technical grade, or better)	67-64-1	200 mL
d_6-acetone (99.9%)	666-52-4	1 mL

Most of the chemicals in the stockroom are ACS grade, but if you need something different, you can find an explanation of chemical grades at the following here:

http://www.reagents.com/products/grades-purity

Future Improvements – The Research Paper

From 3405 Course Syllabus

In writing the proposal, follow the general format of a research paper published in *Inorganic Chemistry* or another of the journals published by the American Chemical Society.

"Starting at the beginning of 2017, all ACS publications will require experimental details to address and emphasize any unexpected, new, and/or significant hazards or risks associated with the reported work."

Ingredients for a Positive Safety Culture DOI: 10.1021/acscentsci.6b00341

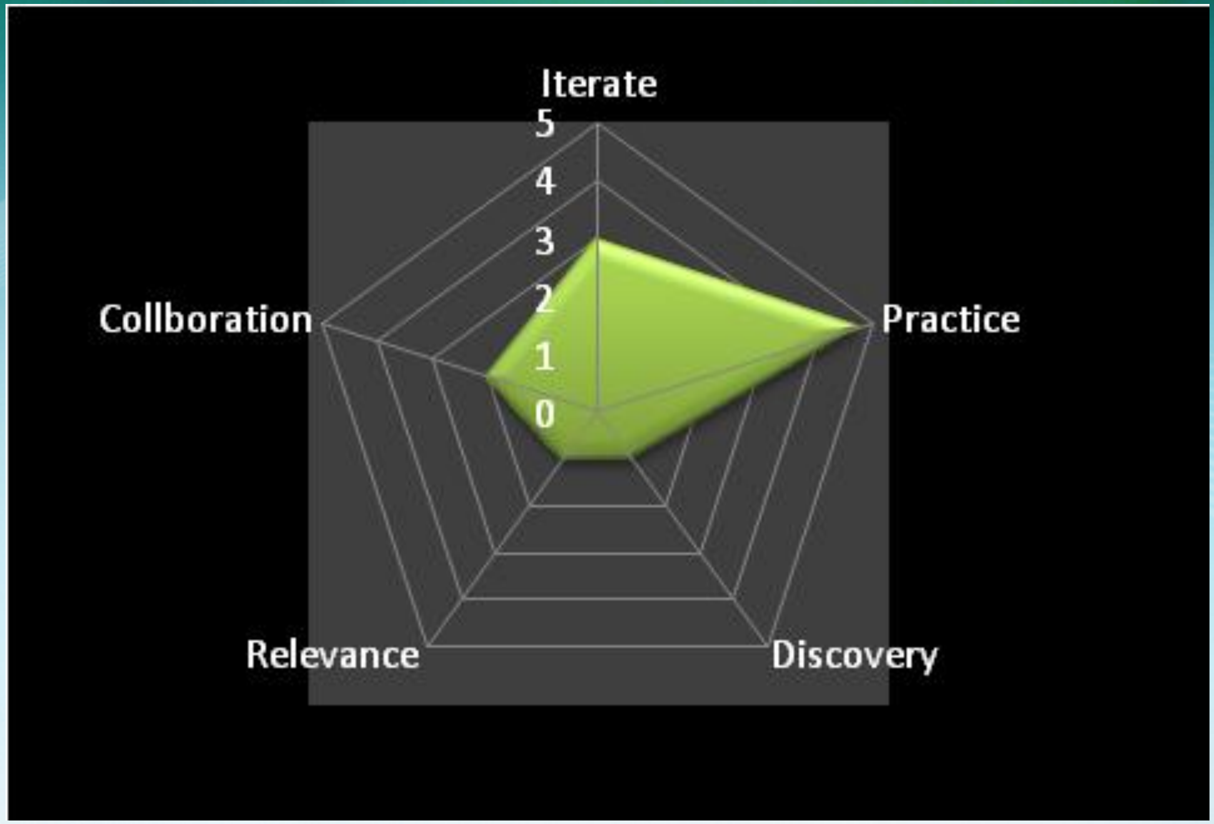
Improve a CURE - Teach Risk Assessment

Recognized Opportunity

Symposium Description

"…Course-based undergraduate research Experiences (CUREs) provide a powerful solution to these challenges by adapting laboratory curricula to a structured, but discovery-based focus. CUREs have been demonstrated to provide many of the same learning gains as traditional research experiences and provide significantly greater capacity for undergraduate research. **This symposium explores successful models at all levels to help interested faculty develop and implement this practice that their home institutions.**"

CURE Aspects Met by Inorganic Research Project



Iterate	3
Practice	4.8
Discovery	1
Relevance	1
Collboration	2

Scientific Practices: Work includes aspects of the scientific method, resilience in research, and dissemination
Discovery: Finding new knowledge beyond what is known by either the students or instructor
Relevance: The work strives for discoveries that are impactful to the discipline or science in general
Collaboration: Students work collaboratively
Iterative: Time is given for students to apply information in designing or improving experiments

Risk Assessment – Necessary in a CURE

- If a CURE is the only research a student participates in, it may be the only time a student has to learn how to ***evaluate disparate chemical safety information***
- In laboratories where students are designing experiments and ***discovery research*** is happening, the hazards will be greater – especially if novel compounds are synthesized
- Increased ***complexity*** of operation increases risk in a teaching laboratory

Risk Assessment – Added Value for a CURE

The process of performing a risk assessment

- Embodies the pedagogy of Course-based Undergraduate Research
- Very likely increases each aspect of the five tenets of CURE (practice, iteration, discovery, relevance, and collaboration)
- Teaches critical thinking – which is not skill, but a method by which we accommodate and assimilate knowledge
- Students begin to understand that safety is integral to the research process, not an added burden

Risk Assessment – Added Value for a CURE

When asked if the process was any "smoother" than previous years, Dr. Wheeler replied that while he was not sure from a safety standpoint, but that he did note that the students were much more organized than previous years

He attributed this to the students having to lay out the synthesis as a series of steps which forced them to critically think about the procedure as compared to just reading a procedure in a paper

Pedagogical Alignment of CUREs and Safety

CURE	SAFE
Cookbook	Rules
Inquiry	Risk
Discovery	Accountability

Acknowledgements

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- The American Chemical Society